(54) METHOD FOR MANUFACTURING BASE MATERIAL FOR OPTICAL FIBER

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[There are no amendments to this patent.]

CLAIMS

1.   A method for manufacturing a base material for an optical fiber, characterized by the fact that in a method for manufacturing a base material for an optical fiber by heating a porous base material having a core-corresponding part and a clad-corresponding part and making it transpartent, it forms a porous base material for the core by depositing fine glass particles generated by supplying a glass raw material into a burner flame by subjecting it to a gas-phase

reaction, transforms it into a porous base material for a core with a raised bulk density by heating said porous base material for the core, and obtains a composite porous base material by depositing fine glass particles for the clad at the outer periphery of said porous base material.

2.   The method for manufacturing a base material for an optical fiber of Claim 1, characterized by the fact that the heating is carried out in a range in which the average bulk density $\bar{\rho}$ (g/cm^3) of the porous base material for the core is $0.4 \leq \bar{\rho} \leq 1.0$.

DETAILED EXPLANATION OF THE INVENTION

INDUSTRIAL APPLICATION FIELD

The present invention pertains to the improvement of a method for manufacturing a base material for an optical fiber by a gas-phase synthesis method.

PRIOR ART

As a method for manufacturing a base material for an optical fiber by a gas-phase synthesis method, a conventional VAD (Vapor-Phase Axial Deposition) method is known. In the method, as shown in Figure 1, a raw material gas for forming a glass is supplied with a combustion gas such as H_2 and O_2 and an inert gas such as Ar to a burner 1 for synthesizing glass fine particles (hereinafter, abbreviated as a burner). As the glass raw material, $SiCl_4$ is usually used, and an additive raw material such as $GeCl_4$ and SiF_4 may also be mixed to adjust the refractive index. As the combustion gas, for example, a hydrocarbon-containing gas such as CH_4, C_2H_6, and C_3H_8, CO, etc., can also be used. Said raw material gas for forming a glass is flame-hydrolyzed in a flame (oxyhydrogen flame in case H_2 and O_2 are used) of the burner 1, so that fine glass particles are generated. The fine glass particles begin to be attached from the tip of a starting rod 2 installed in a rotary lifter 4 and lifted while rotating said starting rod 2, so that a porous base material 3 is formed in the axial direction of the starting rod 2. The porous base material 3 manufactured in this manner is subjected to a heating and dehydrating treatment (hereinafter, called a heating-dehydrating treatment) and a heating and transparency-promoting treatment (hereinafter, called a heating-dehydrating treatment), so that a transparent glass base material is obtained. Said transparent glass base material is transformed into a stretched rod for the core by stretching at a prescribed diameter, and said rod for the core is mounted as a starting material 5 in the rotary lifter 4 in the apparatus constitution as shown in Figure 2. Using a burner 6 for synthesizing the clad, the fine glass particles are deposited at the outer periphery of said rod for the core, so that a composite body composed of the rod for the core and the porous body for the clad is formed. A base material for an optical fiber with the desired clad/core diameter ratio

is obtained by subjecting said composite body to the heating-dehydrating treatment and the heating-transparency treatment similarly to the above case.

PROBLEMS TO BE SOLVED BY THE INVENTION

On the other hand, in the above-mentioned conventional method, the transparent glass base material for the core is transformed into the rod for the core by stretching at a prescribed diameter; however, a flame polishing is required to clean and smooth the outer surface of said rod before depositing the fine glass particles for the clad at the outer periphery of said rod. Usually, an oxyhydrogen flame is used in the flame polishing; however, since the surface of the rod for the core is exposed to the flame, high-concentration OH groups are mixed into the core surface. Furthermore, in the deposition of the fine glass particles for a clad in the subsequent process, since the outer surface of the rod for the core is also heated by the flame of the burner, the OH groups are also mixed into the core. Since the existence of the OH groups markedly degraded the transmission loss, which was an important characteristic of optical fibers, the OH group mixture was a very difficult problem.

The purpose of the present invention is to offer a method that solves the problem of the OH group mixing into the core, and that can manufacture a base material for a high-quality optical fiber with excellent transmission characteristics; the method forms fine glass particles from such a glass raw material by the gas-phase synthesis method, obtains a porous base material by depositing them, and obtains an optical fiber base material by transforming it into a transparent glass.

MEANS TO SOLVE THE PROBLEMS AND OPERATION

The present invention pertains to a method for manufacturing a base material for an optical fiber, characterized by the fact that in a method for manufacturing a base material for an optical fiber by heating a porous base material having a core-corresponding part and a clad-corresponding part, and makes it transparent, it forms a porous base material for a core by depositing fine glass particles generated by supplying a glass raw material into a burner flame by subjecting it to a gas-phase reaction, transforms it into a porous base material for the core with a raised bulk density by heating said porous base material for the core, and obtains a composite porous base material by depositing fine glass particles for the clad at the outer periphery of said porous base material.

As an especially preferred embodiment of the present invention, the heating is carried out in a range in which the average bulk density $\bar{\rho}$ (g/cm^3) of the porous base material for the core is $0.4 \leq \bar{\rho} \leq 1.0$.

In the present invention, the heating-dehydrating treatment after forming the porous base material for the core is similar to the conventional method; however, said dehydrated porous body is heated and shrunk, so that its average bulk density is raised. More preferably, the porous body is heated and shrunk so that the average bulk density $\bar{\rho}$ may be $0.4 \leq \bar{\rho} \leq 1.0$. Such a heating is carried out by maintaining the temperature at 1,200-1,500°C in a He gas atmosphere using an ordinary electric furnace.

In this heated and shrunk state, that is, without a transparency-promoting treatment, stretching, and flame-polishing, the porous base material for the core is mounted as a starting material 5 in a lift rotator 4 with the constitution shown in Figure 2, then fine glass particles for the clad are deposited at its outer periphery by a well-known means using a burner 6 for the clad and grown in the axial direction, so that a composite porous base material composed of the above-mentioned porous base material for the core (starting material 5) and a porous body 7 for the clad is obtained. A transparent glass base material having a core and a clad is obtained by subjecting said composite porous base material to the heating-dehydrating treatment and the heating-transparency-promoting treatment, and it is wire-drawn, so that an optical fiber is obtained.

OPERATION

In the present invention, since the porous base material for the core is not subjected to the transparency-promoting treatment, stretching, and flame-polishing treatment, there is no mixture of OH groups due to the stretching and the flame polishing. Therefore, unlike the conventional method, a high-concentration OH group layer is not formed at the core and clad interface.

Also, as for the porous base material for the core, which is heated and shrunk so that the average bulk density $\bar{\rho}$ may be raised, preferably so that $\bar{\rho}$ is $0.4 \leq \bar{\rho} \leq 1.0$, when fine glass particles for the clad are deposited on it in the next process, since the porous base material is heated by a flame of the burner, OH groups are introduced into the outer periphery. However, after forming the porous body for the clad, the OH groups introduced into the above-mentioned OH groups in the above-mentioned core are removed at the time of the heating-dehydrating treatment of the composite porous base material. At that time, if the average bulk density $\bar{\rho}$ of the porous base material for the core is greater than 1, the OH group removal from the outer surface of the porous base material for the core is insufficient, which is not preferable. Also, if the $\bar{\rho}$ is less than 0.4, cracks are easily generated in the vicinity of the interface with the porous base material for the core when the fine glass particles for the clad are deposited, so that a stable manufacture is difficult, which is not preferable. The average bulk density of the porous body prepared by the ordinary VAD method, etc., is about 0.2-0.3.

According to the present invention, several porous base materials for a core of the clad type fiber were heated and shrunk so that the average bulk density $\bar{\rho}$ had different values, so that a composite porous base material having a core and a clad was obtained. An optical fiber was then obtained by wire-drawing after the transparency treatment, and its OH absorption-loss increase at a wavelength of 1.38 μm was investigated. As for the results, the average bulk density $\bar{\rho}$ (g/cm^3) of the porous base material for the core after heating and shrinking is indicated on the abscissa, and the OH absorption-loss increase (dB/km) of the fiber obtained is indicated on the ordinate. The relationship between them is shown as a graph in Figure 3.

The manufacture of the porous base material by depositing the fine glass particles synthesized in the gas phase from the glass raw material may be carried out by a conventional well-known technique; for example, the VAD method, OVFD method, plasma flame method, etc., can be mentioned.

As the glass raw material for a core or clad, for example, $SiCl_4$, $SiHCl_2$, etc., can be used, and as additives for adjusting the refractive index, etc., for example, $GeCl_4$, SiF_4, SF_6, CCl_2F_5, etc., can be used.

As a fuel gas and a combustion gas, in general H_2, CH_4, C_2H_6, O_2, CO, etc., can be used, and as an inert gas, Ar, N_2, etc., can be used.

The heating-dehydrating treatment of the porous base material for the core is carried out by heating at a temperature of 800-1,100°C in a mixed atmosphere of He gas and a Cl_2 or Cl compound gas using an electric furnace, for instance.

The porous body for a core that is heated and dehydrated is heated at a temperature of 1,200-1,500°C in a He gas atmosphere using the electric furnace as mentioned above, so that the bulk density is raised.

Also, the process for forming the composite porous base material by depositing the fine glass particles for a clad at the outer periphery of the porous base material for the core, of which the bulk density is raised, may be carried out similarly to the formation of the above-mentioned porous body for a core, and the heating-dehydrating treatment and the heating-transparency-promoting treatment of said porous base material may also be similarly carried out.

APPLICATION EXAMPLES

APPLICATION EXAMPLE 1

In the apparatus constitution shown in Figure 1, H_2 at 2.35 L/min, O_2 at 7 L/min, Ar at 4 L/min, $SiCl_4$ at 560 cc/min, and $GeCl_4$ at 32 cc/min were supplied to a burner for the core, and a porous body for the core was manufactured at a lift rate [in the rotator] of 59 mm/min. The

outer diameter of the porous body for the core obtained was 87 mm ϕ, and the average bulk density was 0.19 g/cm^3. Said porous body was heated and dehydrated at a temperature of 1,050°C in a He gas atmosphere containing 6 vol% Cl$_2$ (gas partial pressure ratio) using an electric furnace. Next, it was heated at 1,400°C in an atmosphere of only He gas, so that the average bulk density of said porous base material was controlled to 0.72.

Using the porous base material for a core heated and shrunk as a starting material 5 in the apparatus constitution shown in Figure 2, a porous body for the clad was formed at its outer periphery. At that time, H$_2$ at 38 L/min, O$_2$ at 45 l/min, Ar at 13 L/min, and SiCl$_4$ at 3.20 L/min were supplied to a burner for the clad, and the lift rate was set to 84 mm/min. The bulk density of the porous body part for a clad was 0.31 g/cm^3.

The composite porous base material obtained was heated and dehydrated at a temperature of 1,060°C in a He gas atmosphere containing 6 vol% Cl$_2$ using the electric furnace. Next, it was heated at 1,620°C in an atmosphere of only He gas, so that a transparent glass was obtained. At the core and clad interface of the transparent glass parent material obtained, the generation of bubbles was not seen.

A clad fiber with an outer diameter of 125 μm and a refractive index difference of 0.94% was prepared by wire-drawing said transparent glass base material; when the optical transmission loss was measured, favorable characteristics, i.e., an OH absorption loss of 0.31 dB/km at a wavelength of 1.38 μm and 0.52 dB/km at a wavelength of 1.3 μm were shown.

COMPARATIVE EXAMPLE 1

Similarly to Application Example 1 except for raising the temperature for heating and shrinking a porous base material for the core to 1,600°C, a transparent glass was obtained. Without stretching the rod, fine glass particles for the clad were deposited at its outer periphery under the same conditions as those of Application Example 1, similarly heated and dehydrated, and subjected to a transparent-glass formation treatment, so that a transparent glass base material in which bubbles were generated at part of the interface of the core and the clad was obtained. The part without bubbles of said base material was wire-drawn into a fiber; when its transmission loss was measured, the OH absorption loss was 3.0 dB/km at a wavelength of 1.38 μm and 1.2 dB/km at a wavelength of 1.3 μm. These values were higher than those of the product of the present invention of Application Example 1, and the transmission characteristics were inferior.

APPLICATION EXAMPLE 2

In the apparatus constitution shown in Figure 1, H$_2$ at 2.8 l/min, O$_2$ at 9 L/min, Ar at 5 l/min, and SiCl$_4$ at 350 cc/min were supplied to a burner for the core, and a porous body for the core was manufactured at a lift rate of 80 mm/min. The outer diameter of the porous body for the

core obtained was 55 mm ϕ, and the average bulk density was 0.27. Said porous body was heated and dehydrated at a temperature of 1,050°C in an atmosphere of He at 10 L/min He and Cl_2 at 500 cc/min using an electric furnace. Next, it was heated and shrunk at 1,460°C in an atmosphere of only He at 10 L/min, so that the average bulk density was increased to 0.92.

As for said porous base material that was heated and shrunk, in the apparatus constitution shown in Figure 2, H_2 at 44 L/min, O_2 at 62 L/min, Ar at 15 L/min, and $SiCl_4$ at 7.4 L/min were supplied to a burner for the clad, and fine glass particles for the clad were deposited at a lift rate set to 71 mm/h, so that a composite porous body with an outer diameter of 210 mm ϕ was obtained. The composite porous base material was heated and dehydrated at a temperature of 1,040°C in a He gas atmosphere containing 6 vol% Cl_2 using the electric furnace. Next, it was heated at 1,250°C in a He gas atmosphere containing 2.7 vol% SiF_4, then F was added. This was heated at 1,610°C in a He gas atmosphere containing 2.7 vol% SiF_4, so that a transparent glass was formed.

After heating and stretching the glass rod obtained, fine glass particles for the clad were deposited again under conditions similar to the above-mentioned conditions, so that the clad/core diameter ratio was 15, and a transparent glass was obtained similarly to the above.

An optical fiber with an outer diameter of 125 μm was obtained by wire-drawing the base material rod for an optical fiber obtained in this manner. Said fiber was a single-mode fiber in which the refractive index difference of the SiO_2 clad added was 0.29%. It showed good characteristics, in which the cut-off wavelength λ was 1.17 μm, the transmission loss was 0.34 dB/km at a wavelength of 1.3 μm and 0.19 dB/km at a wavelength of 1.55 μm, and the OH absorption loss was 0.9 dB/km at a wavelength of 1.38 μm.

COMPARATIVE EXAMPLE 2

Similarly to Application Example 2 except for raising the temperature for heating and shrinking the porous base material for the core to 1,630°C, a transparent glass was obtained. Under the same conditions as those in Application Example 2, a base material rod for an optical fiber was obtained and drawn into a fiber. The OH absorption loss of said fiber was as large as 2.9 dB/km at a wavelength of 1.38 μm, and the transmission loss at a wavelength of 1.3 μm was 1.1 dB/km. These characteristics were considerably inferior to those of the product of the present invention of Application Example 2.

EFFECT OF THE INVENTION

According to the present invention, without the heating, stretching, and flame-polishing of a porous body for a core resulting in the mixing of OH groups, said porous body is heated to raise the average bulk density, and a porous body for a clad is formed at its outer periphery.

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Thus, the OH group mixing problem during the clad formation can also be solved.

Therefore, in the present invention, the OH absorption loss is reduced and this method is very favorable for manufacturing the base material for an optical fiber, which can manufacture an optical fiber with excellent transmission characteristics. Also, in the present invention, treatment processes of heating and transparency promotion, stretching, and flame-polishing of the porous body for the core can be omitted, which is industrially advantageous.

BRIEF DESCRIPTION OF THE FIGURES

Figure 1 is an outlined cross section for explaining an embodiment of the processes for manufacturing a porous body for the core of the present invention.

Figure 2 is an outlined cross section for explaining an embodiment of the process for manufacturing a composite porous body by forming a porous body for a clad at the outer periphery of the porous body for the core of the present invention.

Figure 3 is a graph showing the relationship between the average bulk density (g/cm^3) of the porous base material for the core heated and shrunk and the OH absorption loss (dB/km) of a fiber obtained from said base material.
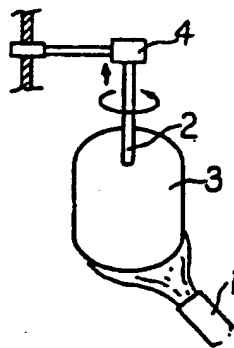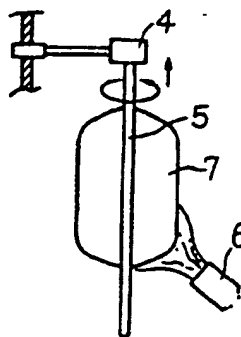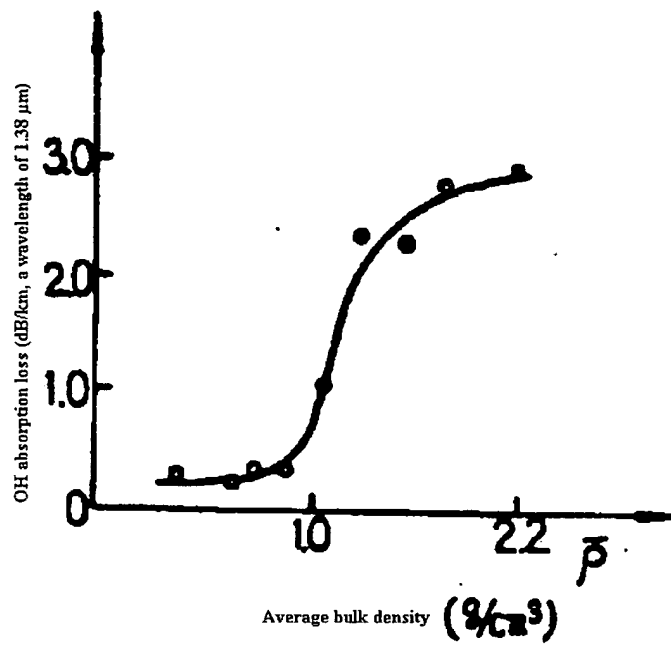

Figure 1

Figure 2

Figure 3

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�554発明の名称　　光フアイバ用母材の製造方法

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明　　細　　書

1. 発明の名称

光フアイバ用母材の製造方法

2. 特許請求の範囲

(1) コア相当部及びクラッド相当部を有する多孔質母材を加熱して透明化することにより光フアイバ用母材を製造する方法において、ガラス原料をバーナ火炎中に供給して気相反応させることにより生成したガラス微粒子を堆積させてコア用多孔質母材を形成し、次に該コア用多孔質母材を加熱することによりかさ密度の上昇したコア用多孔質母材とした後、該コア用多孔質母材の外周部にクラッド用ガラス微粒子を堆積させることにより複合多孔質母材を得ることを特徴とする光フアイバ用母材の製造方法。

(2) 加熱によりコア用多孔質母材の平均かさ密度 $\bar{\rho}$（g/cm^3）を $0.4 \leq \bar{\rho} \leq 1.0$ として行なう特許請求の範囲第1項に記載される光フアイバ用母材の製造方法。

3. 発明の詳細な説明

〔産業上の利用分野〕

本発明は気相合成法による光フアイバ用母材の製造方法の改良に関するものである。

〔従来の技術〕

気相合成法により光フアイバ用母材を製造する方法として、従来VAD（Vaporphase Axial Deposition 気相軸付け）法が知られている。この方法は第1図に示すように、ガラス微粒子合成用バーナ（以下バーナと略する）1にガラス形成原料ガスを、H_2，O_2 等の燃焼ガス及び Ar 等の不活性ガスと共に供給する。ガラス原料としては一般的に $SiCl_4$ が用いられ、また屈折率を調整するために例えば $GeCl_4$，SiF_4 等の添加剤原料を混合してもよい。燃焼ガスとしては例えば CH_4，C_3H_8，C_2H_2 等の炭化水素系ガスや CO 等を用いることもできる。バーナ1の火炎（H_2 及び O_2 を用いた場合は酸水素炎）中で該ガラス形成原料ガスは火炎加水分解反応して、ガラス微粒子を生成する。この

ガラス微粒子を回転引上装置4に取りつけた出発棒2の先端部から付着させ始め、該出発棒2を回転させつつ引き上げてゆくことにより、多孔質母材3を出発　2の軸方向に形成してゆく。上記で作製された多孔質母材3について、次に加熱して脱水する処理（以下加熱脱水処理と称す）及び加熱して透明化する処理（以下加熱透明化処理と称す）を施こすことにより、透明ガラス母材を得る。該透明ガラス母材を所定径に延伸してコア用延伸ロッドとした後、第2図に示すような装置構成において該コア用ロッドを出発材5として回転引上装置4に装着し、クラッド合成用バーナ6を用いて該コア用ロッドの外周にガラス微粒子を堆積して、コア用ロッドとクラッド用多孔質体からなる複合体を形成する。該複合体を前記したと同様に加熱脱水処理及び加熱透明化処理して、所望のクラッド／コア径比を有する光フアイバ用母材を得る。

[発明が解決しようとする問題点]

ところで上記した従来法では、コア用透明ガラス母材を所定径に延伸してコア用ロッドとするが、該コア用ロッドの外周にクラッド用ガラス微粒子を堆積させる前に、該ロッドの外表面を清浄、平滑にする目的で、火炎研磨を実施する必要がある。通常この火炎研磨には酸水素炎が用いられるが、コア用ロッドの表面が火炎に曝されるため、コア表面に高濃度のOH基が混入してしまう。さらに続く工程のクラッド用ガラス微粒子堆積に際しても、クラッド用バーナの火炎によりコア用ロッド外表面は加熱されるので、これによってもコアにOH基が混入してしまう。OH基の存在は光フアイバの重要な特性である伝送損失を著るしく劣化させるので、このOH基混入は非常に困つた問題であつた。

本発明はこの種のガラス原料から気相合成法によりガラス微粒子を形成し、これを堆積して多孔質母材を得て、これを透明ガラス化して光フアイバ母材を得る方法における、コアへのOH基混入の問題を解消して、伝送特性に優れた高品質の光フアイバ用母材を製造できる方法を提供することを目的とするものである。

[問題点を解決するための手段・作用]

本発明はコア相当部及びクラッド相当部を有する多孔質母材を加熱して透明化することにより光フアイバ用母材を製造する方法において、ガラス原料をバーナ火炎中に供給して気相反応させることにより生成したガラス微粒子を堆積させてコア用多孔質母材を形成し、次に該コア用多孔質母材を加熱することによりかさ密度の上昇したコア用多孔質母材とした後、該コア用多孔質母材の外周部にクラッド用ガラス微粒子を堆積させることにより複合多孔質母材を得ることを特徴とする光フアイバ用母材の製造方法に関する。

本発明の特に好ましい実施態様としては、加熱によりコア用多孔質母材の平均かさ密度 $\bar{\rho}$（ g/cm^3 ）を $0.4 \leq \bar{\rho} \leq 1.0$ として行なう上記方法が挙げられる。

本発明はコア用多孔質母材を形成して加熱脱水処理を行うところまでは従来と同様であるが、該脱水処理された多孔質体を加熱して収縮させ、その平均かさ密度を高める。特に好ましくはその平均かさ密度 $\bar{\rho}$ が、 $0.4 \leq \bar{\rho} \leq 1.0$ となるように加熱して収縮させる。このような加熱は通常電気炉を用いてHe ガス雰囲気下温度1200°～1500℃に保持することにより行われる。

加熱収縮されたコア用多孔質母材を、この状態のままで、すなわち透明化処理や延伸や火炎研磨なしに、第2図の構成の引上回転装置4に出発材5として装着して、クラッド用バーナ6を用いて公知の手段によりその外周部にクラッド用ガラス微粒子を堆積させて軸方向に成長させ、前記コア用多孔質母材（出発材5）とクラッド用多孔質体7とからなる複合多孔質母材を得る。該複合多孔質母材について、加熱脱水処理及び加熱透明化処理を行うことによりコア・クラッドを有する透明ガラス母材を得、またこれを線引きすることにより光フアイバが得られる。

[作用]

本発明はコア用多孔質母材についての透明化処理、延伸及び火炎研磨処理を行わないので、延伸や火炎研磨によるOH基の混入がない。従つて、従来法のようにコア・クラッド界面に高濃度のOH基層が形成されることはない。

また、平均かさ密度$\bar{\rho}$を高め、特に好ましくは$\bar{\rho}$が$0.4 \leq \bar{\rho} \leq 1.0$となるよう加熱収縮されたコア用多孔質母材は、次の工程でクラッド用ガラス微粒子を堆積させる際にクラッド用バーナの火炎で加熱されるために、その外周囲にOH基が取り込まれるが、クラッド用多孔質体を形成した後、複合多孔質母材の加熱脱水処理の際に上記のコア中に取り込まれたOH基を除去される。このときコア用多孔質母材の平均かさ密度$\bar{\rho}$が1より大であるとコア用多孔質母材の外表面のOH基除去が不充分となり好ましくない。また$\bar{\rho}$が0.4未満ではクラッド用ガラス微粒子を堆積する際に、コア用多孔質母材との界面付近でクラックが発生し易くなり、安定な製造が困難となるため好ましくない。なお、通常のVAD法等により作成した多孔質体の平均かさ密度は0.2〜0.3程度である。

本発明により、グレーデッド型ファイバのコア用多孔質母材の複数について、その平均かさ密度$\bar{\rho}$が種々の値となるように加熱収縮した後に、コア・クラッドを有する複合多孔質母材とし、透明化後線引きして得たファイバの波長1.38μmにおけるOH吸収ロス増を調べた。得られた結果を、加熱収縮後のコア用多孔質母材の平均かさ密度$\bar{\rho}$(g/cm^3)を横軸に、得られたファイバのOH吸収ロス増(dB/km)を縦軸にとつて両者の関係を示したグラフとして、第3図に示す。

本発明における、ガラス原料から気相合成したガラス微粒子を堆積することによる多孔質母材の製造は従来公知の技術にて行えばよく、例えばVAD法、OVPD法、プラズマ火炎法等を挙げることができる。

コア又はクラッド用のガラス原料としては例えば$SiCl_4$, $SiHCl_3$等を用いることができ、これ等に屈折率その他を調整するための添加剤として例えば$GeCl_4$, SiF_4, SF_6, CCl_2F_2等を加えることができる。

燃料ガス及び助燃ガスとしては一般的なH_2, CH_4, C_3H_8, O_2, CO等を用いることができ、不活性ガスとしてはAr, N_2等を用いることができる。

コア用多孔質母材の加熱脱水処理は、例えば電気炉を用いて通常HeガスとCl_2又はCl化合物ガスとの混合雰囲気下温度800〜1100℃にて加熱することによる。

加熱脱水処理したコア用多孔質体は次に前記したように電気炉を用いて通常はHeガス雰囲気下で温度1200〜1500℃にて加熱して、カサ密度を上昇させる。

またカサ密度を上昇させたコア用多孔質母材の外周部にクラッド用ガラス微粒子を堆積させ複合多孔質母材とする工程は、上記したコア用多孔質体形成と同様に行なえばよく、該複合多孔質母材の加熱脱水処理、加熱透明化処理も同様に行なえばよい。

[実施例]
実施例1

第1図に示す装置構成において、コア用バーナにH_2 235l/分、O_2 7l/分、Ar 4l/分、$SiCl_4$ 560cc/分及び$GeCl_4$ 32cc/分を供給して、引き上げ速度59mm/時でコア用多孔質体を作製した。得られたコア用多孔質体の外径は87$mm\phi$、平均かさ密度は0.19g/cm^3であつた。該コア用多孔質体を電気炉を用いて、温度1050℃でCl_2 6容積%(ガス分圧比)を含むHeガス雰囲気中にて加熱脱水処理した。次にHeガスのみの雰囲気として、1400℃に加熱することにより、該多孔質母材の平均かさ密度を0.72とした。

この加熱収縮したコア用多孔質母材を第2図に示す装置構成において出発材5とし、その外周にクラッド用多孔質体を形成した。このときクラッド用バーナにはH_2 38l/分、O_2 45l/分、Ar 13l/分、$SiCl_4$ 520l

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/分を供給し、引上速度は64mm/時とした。クラッド用多孔質体部分のかさ密度は0.31g/cm³であった。

得られた複合多孔質母材を、電気炉を用いてCl₂ 6容積%を含むHe ガス雰囲気中にて1060℃で加熱脱水処理した。次にHe のみのガス雰囲気として1620℃に加熱して透明ガラス化したところ、得られた透明ガラス母材のコア・クラッド界面には気泡の発生は見られなかった。

該透明ガラス母材を線引きして外径125μm、屈折率差0.94%のグレーデッド型ファイバを作製し、その光伝送損失を測定したところ、波長1.38μm におけるOH 吸収ロスは0.31dB/km、波長1.3μm では0.52dB/kmと良好な特性を示した。

比較例1

実施例1において他の条件は同様にしてコア用多孔質母材を加熱収縮させる温度を1,600℃に上げたところ、透明ガラス化した。このロッドを延伸することなく、その外側に実施例1

かさ密度を0.92に増加させた。

該加熱収縮した多孔質体について第2図の装置構成で、クラッド用バーナにH₂ 44ℓ/分、O₂ 62ℓ/分、Ar 15ℓ/分、SiCl₄ 7.4ℓ/分を供給し、引上速度71mm/時で、クラッド用ガラス微粒子体を堆積させて、外径210mm¢の複合多孔質体を得た。この複合多孔質体を電気炉を用いて、Cl₂ 6容積%を含むHe ガス雰囲気下1060℃で加熱脱水処理し、続いて27容積%のSiF₄ を含むHe ガス雰囲気下1250℃に加熱してF添加し、次いで27容積%のSiF₄ を含むHe 雰囲気にて1610℃に加熱することにより透明ガラス化した。

得られたガラスロッドを加熱延伸した後、クラッド/コア径比が15となるように再度上記と同様の条件にてクラッド用ガラス微粒子を堆積し、上記同様に透明ガラス化した。

以上で得られた光ファイバ用母材ロッドを線引きして外径125μm の光ファイバとした。該ファイバは純シリカのコアとF（ふっ素）が添

と同条件でクラッド用ガラス微粒子を堆積して同様に加熱脱水処理し、次に透明ガラス化処理を行ったところ、コアとクラッドガラスの界面の一部に気泡の発生した透明ガラス母材が得られた。該母材の気泡のない部分を線引きしてファイバとし、その伝送ロスを測定したところ、波長1.38μm においてOH 吸収ロスは3.0dB/km、波長1.3μm で1.2dB/kmと実施例1の本発明品に比して高い値であり、伝送特性が劣っていた。

実施例2

第1図の装置構成にて、コア用バーナにH₂ 28ℓ/分、O₂ 9ℓ/分、Ar 5ℓ/分及びSiCl₄ 350cc/分を供給して、引上速度80mm/分で、外径55mm¢、平均カサ密度0.27のコア用多孔質体を作製した。該コア用多孔質体を電気炉にて、He 10ℓ/分、Cl₂ 500cc/分の雰囲気下、温度1050℃にて加熱脱水処理した後、He 10ℓ/分のみの雰囲気とし、1460℃で加熱収縮処理して、その平均

加されたSiO₂ クラッドの屈折率差が0.29%であるシングルモードファイバであり、カットオフ波長λ=1.17μm 、伝送ロスは波長1.3μm で0.34dB/km、波長1.55μm で0.19dB/km、OH 吸収ロスは波長1.38μm で0.9dB/kmと良好な特性を示した。

比較例2

実施例2において、コア用多孔質体の加熱収縮処理温度を1630℃に上げて透明ガラス化した以外は、実施例2におけると同条件にて行って光ファイバ用母材ロッドを得て、これを線引きしファイバとした。該ファイバのOH 吸収ロスは波長1.38μm で29dB/kmと非常に大きくなり、また波長1.3μm での伝送ロスも1.1dB/kmであり、実施例2の本発明品に比して著しく劣化していた。

〔発明の効果〕

本発明は、OH 基混入の原因となるコア用多孔質体の加熱延伸、火炎研磨を行なわずに、該多孔質体の平均カサ密度を上昇せしめるよう加

熱収縮した後、この外周にクラッド用多孔質体を形成することで、クラッド形成時でのOH 基混入の問題をも解決できる。

したがつて本発明はOH 吸収ロスが低減されており、かつ伝送特性に優れた光フアイバを製造できる光フアイバ用母材の製造方法として非常に有利である。またコア用多孔質体の加熱透明化、延伸、火炎研磨といつた処理工程を省略できる点も工程的に有利である。

4. 図面の簡単な説明

第1図は本発明のコア用多孔質体を作製する工程の実施態様を説明する概略断面図、

第2図は本発明のコア用多孔質体外周にクラッド用多孔質体を形成し複合多孔質を作製する工程の実施態様を説明する概略断面図、

第3図は加熱収縮されたコア用多孔質母材の平均かさ密度(g/cm^3)と、該母材から得たフアイバのOH 吸収ロス(dB/km)の関係を示すグラフである。

第1図

第2図

第3図
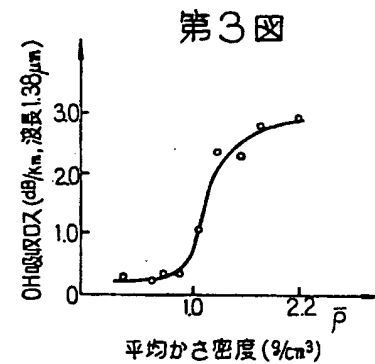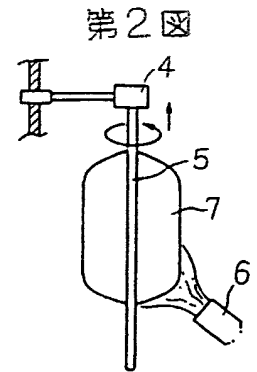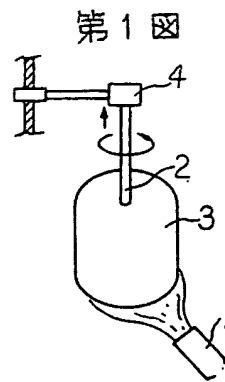
第1頁の続き

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